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Pyrolysis of cassava rhizome in a counter-rotating twin screw reactor unit

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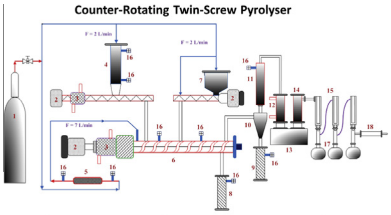
HIGHLIGHTS

- A counter-rotating twin screw pyrolysis reactor was investigated.
- The effects of temperature, biomass particle size and carrier gas were studied.
- Pyrolysis product yields and bio-oil properties were monitored.
- The optimum pyrolysis temperature was 550 °C.
- The maximum bio-oil yield was 50 wt.% on dry biomass basis.

GRAPHICAL ABSTRACT



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ABSTRACT

A counter-rotating twin screw reactor unit was investigated for its behaviour in the pyrolysis of cassava rhizome biomass. Several parameters such as pyrolysis temperature in the range of 500–700 °C, biomass particle size of <0.6 mm, the use of sand as heat transfer medium, nitrogen flow rate of 4–10 L/min and nitrogen pressure of 1–3 bar were thoroughly examined. It was found that the pyrolysis temperature of 550 °C could maximise the bio-oil yield (50 wt.%). The other optimum parameters for maximising the bio-oil yield were the biomass particle size of 0.250–0.425 mm, the nitrogen flow rate of 4 L/min and the nitrogen pressure of 2 bar. The use of the heat transfer medium could increase the bio-oil yield to a certain extent. Moreover, the water content of bio-oil produced with the counter-rotating twin screw reactor was relatively low, whereas the solids content was relatively high, compared to some other reactor configurations.

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1. Introduction

The current world dependency of fossil fuels is reflecting the looming crises of global warming. One way to mitigate this is to promote the use of alternative fuels such as biomass. Biomass is known as a renewable, sustainable and environmentally benign fuel. There are several types of lignocellulosic biomass materials. One type that is of great attention is agricultural waste such as cassava residues. In 2012, the world production of cassava crop was around 230 million tons (FAOSTAT, 2012), whereas in Thailand its production was around 22 million tons. According to the 2011 Thailand alternative energy report (DEDE, 2011), the cassava stalk and rhizome residues were 978.41 and 760.37 thousand tons of oil equivalent. Part of the cassava stalk was set aside for re-plantation, whereas the rhizome is normally left as residue. Therefore, the exploitation of this biomass would be beneficial to the whole world including Thailand.

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Biomass can be directly used by traditional combustion technology or converted into a more flexible fuel such as bio-oil by pyrolysis technology. The bio-oil is advantageous as its energy density is 4–5 times higher than the solid biomass (Zheng, 2007). The bio-oil may be used as burner fuel oil. Recently, Bridgwater (2012) stated that in fast pyrolysis of biomass for bio-oil production, the pyrolyser or pyrolysis reactor is regarded as a key part. The reactor types also affect the yield and properties of bio-oil. There are many types of reactor designed and tested in pyrolysis of biomass such as free-fall, fluidised-bed, vacuum, ablative, rotating cone and auger. Brown and Holmgren (2009) compared several type of reactors and stated that the auger reactor had high potential in technical and market aspects since this design is relatively simple, applies low carrier gas flow and is suitable for large biomass particles.

Previous studies investigated two types of auger reactor, namely single screw (Garcia-Perez et al., 2007; Ingram et al., 2007; Kim et al., 2011; Liaw et al., 2012, 2013; Pittman et al., 2012; Puy et al., 2011; Tripathi et al., 2010; Zheng et al., 2012; Zhou et al., 2013a) and twin screw (Brown and Brown, 2012; Raffelt et al., 2006), in pyrolysis of biomass. The majority of the work focused on single screw reactors as they are simpler in design and operation. Several types of feedstock were pyrolysed in single screw reactors such as pine wood (Hassan et al., 2009; Hilten et al., 2009; Ingram et al., 2007; Puy et al., 2011; Thangalazhy-Gopakumar et al., 2010), pine bark (Ingram et al., 2007; Tripathi et al., 2010), pine lumber (Tripathi et al., 2010), oak wood and oak bark (Ingram et al., 2007; Tripathi et al., 2010), Douglas fir wood (Liaw et al., 2012; Zhou et al., 2013b), corn stalk (Pittman et al., 2012), *Gmelina arborea* (Okoroigwe et al., 2012), switch grass (Kim et al., 2011), sewage sludge (Samanya et al., 2012), and deinking sludge (Yang et al., 2013) at 454–600 °C. The bio-oil yields were in the range of 46.8–60% (Garcia-Perez et al., 2007; Ingram et al., 2007; Kim et al., 2011; Liaw et al., 2012, 2013; Pittman et al., 2012; Puy et al., 2011; Tripathi et al., 2010; Zheng et al., 2012; Zhou et al., 2013a). To improve the heat transfer, twin screw reactor were designed and tested with wood and straw (Brown and Brown, 2012; Raffelt et al., 2006) at temperatures of 425–600 °C. The bio-oil yields obtained with the twin screw reactors were in the range of 42–74%, which was somewhat higher than those of the single screw reactors. The reason for this could be related to the higher heat transfer or heating rate of the twin-screw reactors. For both types of auger reactors, the process parameters investigated were reaction temperature (Kim et al., 2011; Liaw et al., 2012, 2013; Pittman et al., 2012; Puy et al., 2011; Zhou et al., 2013a,b), solid residence time (Puy et al., 2011), biomass feed rate (Puy et al., 2011), heat carrier inlet temperature (Brown and Brown, 2012), heat carrier mass flow rate (Brown and Brown, 2012), rotational speed of screw (Brown and Brown, 2012) and volumetric flow rate of sweep gas (Brown and Brown, 2012). However, the influences of these parameters in twin screw reactors were monitored only on bio-oil yield, whereas the bio-oil properties were not reported.

To further develop the design of auger reactors, the two screws were designed in the current work to rotate on the opposite direction. This type of reactor is referred to as a "counter-rotating twin screw". It is the purpose of this work to examine the behavior of the counter-rotating twin screw reactor in the production of bio-oil from a selected biomass feedstock, which is cassava rhizome. This biomass material was previously tested in fast pyrolysis process applying a fluidised-bed reactor (Pattiya and Suttibak, 2012) and a free-fall reactor (Pattiya et al., 2012). This paper also focuses on the influence of pyrolysis temperature, biomass particle size, heat transfer medium, sweep gas flow rate and carrier gas pressure on product distribution, as well as the characterisation of the obtained bio-oil.

2. Methods

2.1. Biomass feedstock

Cassava rhizome obtained from north-east Thailand was investigated in this work. The fresh biomass sample was first dried by sunlight reaching the moisture content of approximately 15 wt.%. It was further dried in an oven at 105 °C for 48 h to obtain a bone-dry sample. The dried sample was sieved into a particle size range of 0.250–0.425 mm. The sample was then characterised for their proximate, ultimate and heating value analyses. The proximate analysis was carried out to determine the contents of moisture, volatile, fixed carbon and ash according to the ASTM E1756-01, E872-82 and E1755-01 standard methods. The ultimate analysis was carried out using Leco CHN/S determinators analyser according to ISO/IEEC guide 22 and EN 45014 methods.

The higher heating value (HHV) of the biomass sample was calculated from the ultimate analysis results using the correlation developed by Sheng and Azevedo (2005) as shown by the following equation:

$$\text{HHV (MJ/kg)} = -1.3675 + 0.3137\text{C} + 0.7009\text{H} + 0.0318\text{O}^* \quad (1)$$

where C, H and Ash are percentages of carbon, hydrogen and ash on dry basis and O^* is calculated from:

$$\text{O}^* = 100 - \text{C} - \text{H} - \text{Ash} \quad (2)$$

The lower heating value (LHV) was calculated from the following equation (ECN, 2005):

$$\text{LHV (MJ/kg)} = \text{HHV} - 2.442 \times 8.936\,\text{H}/100 \quad (3)$$

The characterisation results of the cassava rhizome sample were summarised in Table 1.

2.2. Pyrolysis apparatus

Production of bio-oil from cassava rhizome was performed using a counter-rotating twin screw pyrolysis reactor unit (Fig. 1) The unit was composed mainly of a biomass hopper, a sand hopper, biomass and sand screw feeders, a nitrogen pre-heater, an auger or twin screw reactor, a solids canister, a cyclone separator, a char pot, a hot filter and a bio-oil product collection system. The biomass hopper of 6.5 L capacity and the sand hopper of 1.5 L capacity were made of 304 stainless steel material. The biomass screw feeder was driven by a DC motor at a speed of 9 rpm and could deliver a biomass feed rate of 380–400 g/h. The sand screw feeder driven by a DC motor at a speed of 27 rpm could feed the sand of 0.60–

Table 1
Main characteristics of cassava rhizome.

Analysis	This work	Hilten et al. (2009)	Pattiya (2011)
Proximate (wt.%, dry basis)			
Volatile matter	81.9	77.8	81.5
Fixed carbon[a]	10.7	18.2	14.9
Ash	7.3	4.1	3.6
Ultimate (wt.%, dry, ash-free basis)			
Carbon	53.4	51.6	51.4
Hydrogen	7.6	6.7	7.3
Nitrogen	1.1	1.3	1.0
Sulfur	<0.1	<0.1	<0.1
Oxygen[a]	38.0	40.5	40.2
H/C molar ratio	1.71	1.50	1.70
O/C molar ratio	0.53	0.60	0.60
Molecular formula	$CH_{1.71}O_{0.53}$	$CH_{1.54}O_{0.59}$	$CH_{1.71}O_{0.59}$
Heating value (MJ/kg, dry basis)			
HHV	21.7	19.9	20.3
LHV	20.0	18.5	18.8

[a] Calculated by difference.

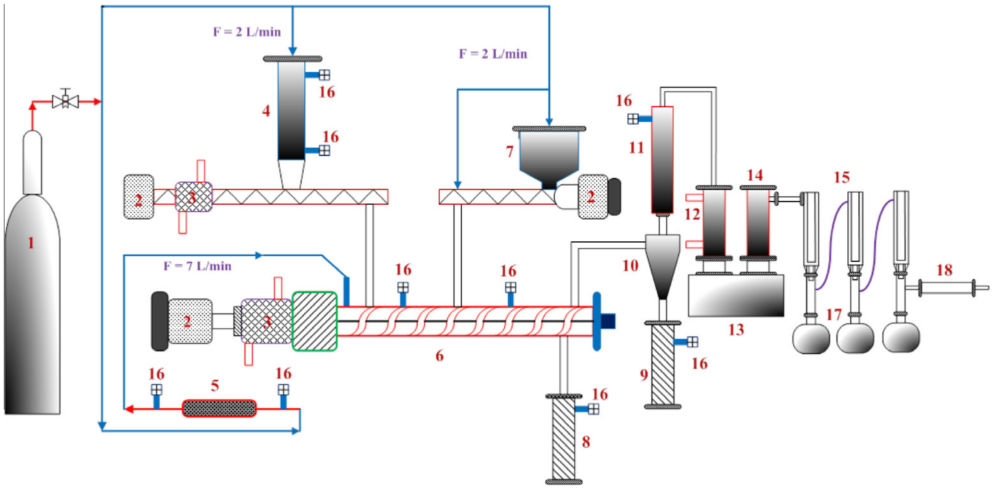

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Fig. 1. Schematic diagram of the counter-rotating twin-screw pyrolysis apparatus.

0.85 mm particle size at a feed rate of 2 kg/h. The nitrogen pre-heater was made from a 304 stainless steel pipe of 150 mm length and 38 mm ID. The pre-heater pipe was filled with steel balls of 3 mm OD. The twin screw auger reactor was 450 mm long and was composed of an omega-shaped vessel made from two 304 stainless steel pipes of 44 mm ID and 3 mm thickness cut and welded together and two screws of 25 mm OD. The solids canister, cyclone separator and char pot were made from 304 stainless steel tubes. The hot filter was made from a 304 stainless steel tube of 300 mm high and 37 mm ID. Inside the hot filter was a fixed bed of approximately 5 g of glass wool. The bio-oil product collection system was composed of a water-cooled condenser, an electrostatic precipitator (ESP), a bio-oil storage tank, three dry ice/acetone condensers, three bio-oil pots and a cotton wool filter.

2.3. Pyrolysis experiments

Pyrolysis experiments were carried out on cassava rhizome to investigate the effect of pyrolysis temperature, biomass particle size, heat transfer medium and carrier gas flow rate and pressure on yields of pyrolysis products. Each experiment was initiated by filling 300 g of the biomass sample of <0.250, 0.250–0.425 or 0.425–0.600 mm particle size into the biomass hopper and 2 kg of 0.425–0.600 mm silica sand into the sand hopper. Subsequently, the nitrogen pre-heater, auger reactor, sand hopper, solids canister, cyclone separator, char pot, hot filter and hot filter-to-condenser line were heated using electrical heaters. The temperature of the nitrogen pre-heater was set at 600 °C in order to obtain the twin screw reactor inlet nitrogen temperature of around 400 °C. The auger reactor temperature was varied from at 500 to 700 °C, whereas the temperature of the sand hopper was fixed at 500 °C. All other heated parts of the unit were controlled at 420 °C. The nitrogen flow rates at the sand hopper top and biomass hopper top were kept constant at 2 L/min, while the nitrogen flow rate at the auger reactor was varied from 4 to 7 L/min. The pressure of the nitrogen was 1–3 bar. Once all temperatures were at steady state, the biomass and hot sand were continuously fed into the reactor where pyrolysis reaction took place, producing pyrolysis vapour and solid char. The coarse char particles together with sand exit the reactor at the end and dropped into the solids canister, whereas the pyrolysis vapour and some char fines were entrained out of the reactor passing the cyclone and the glass wool hot filter where most of the char fines were captured. The vapour was then condensed into bio-oil and collected at the bio-oil storage tank and bio-oil pot.

2.4. Analysis of Bio-oil

2.4.1. Water content

The water content of the bio-oil products was analysed according to ASTM E203 by volumetric Karl Fischer titration using Mettler Toledo V20 automatic titrator. The titration reagent used was HYDRANAL Composite 5 K and the titration solvent was HYDRANAL Working Medium K. The titration was performed at least in triplicate. This was performed at the Center of Scientific and Technological Equipment (CSTE), Suranaree University of Technology, Nakhon Ratchasima, Thailand.

2.4.2. Solids content

The solids content of bio-oil was defined as ethanol insoluble matter and determined by vacuum filtration technique. About 2–3 g of bio-oil was dissolved in ethanol and filtered through a pre-dried and pre-weighed Whatman No. 3 qualitative filter paper with mean pore size of 6 μm. The liquid was then washed with an excess amount of ethanol until the filtrate was clear to ensure that there was no organic liquid left on the paper. The filter paper with the solid was air-dried for approximately 15 min and further dried in an oven at 105 °C for 30 min. Subsequently, the paper was cooled in a desiccator and weighed. This method was recommended by Oasmaa and Peacocke (2010).

2.4.3. pH value

The pH value of bio-oil was measured by a pH meter (COMBI pH/mV/Temp Bench Meter) at room temperature. The instrument was calibrated with liquid calibration standards of pH 4 to 7 prior to the measurement.

2.4.4. Density

The density of bio-oil was the weight of bio-oil contained in a unit volume (g/ml), which was measured by a density bottle at room temperature (about 30 °C).

2.4.5. Heating value

The HHV of the bio-oil samples were measured using an Gallenkamp auto bomb oxygen bomb calorimeter following the ASTM D4809 standard method at the Center for Scientific and Technological Equipment (CSTE), Suranaree University of Technology, Thailand.

2.4.6. Viscosity

The viscosity of bio-oil in mm^2/s (cSt) was measured at 40 °C using a Cannon–Fenske Routine Viscometer (size 450).

2.4.7. Ash content

The ash content of bio-oil was indicated as the amount of residues after heating bio-oil to 775 °C with oxygen supply for 24 h. Direct heating of bio-oil would result in foaming and splashing caused by their high water content. Therefore, the first controlled evaporation of water at 105 °C was conducted before rapid heating to 775 °C (Lu et al., 2009; Oasmaa and Peacocke, 2010).

3. Results and discussion

3.1. Effect of pyrolysis temperature on product distribution

To investigate the effect of pyrolysis temperature on product distribution, cassava rhizome of a particle size range of 0.250–0.425 mm was pyrolysed in the counter-rotating twin screw reactor at 500–700 °C using sand as heat carrier and nitrogen at 1 bar and 7 L/min as sweep gas. The result is illustrated by Fig. 2. It can be seen that the pyrolysis temperature significantly affected the yields of bio-oil, char and permanent gas. The yield of bio-oil increased when increasing the temperature from 500 to 550 °C and reached a maximum of approximately 50 wt.%. When further increasing the temperature from 550 to 700 °C, the bio-oil yield dropped, whereas the gas yield increased accordingly. This is possibly due to the thermal cracking of the pyrolysis vapour. Fig. 2 also shows that the char yield linearly decreased with pyrolysis temperature. This is possibly due to the increased thermal decomposition of the solid biomass/char at elevated temperature. When comparing the bio-oil yield in this work with those obtained from pyrolysis of red oak (Brown and Brown, 2012), wood sawdust, wheat straw chops and rice straw chops (Raffelt et al., 2006), in a co-rotating twin screw reactor, it was found that the maximum yield of cassava rhizome bio-oil from the counter-rotating reactor was lower than that of the red oak, but was comparable to that of the wheat or rice straw chops bio-oils. Cassava rhizome was previously pyrolysed in fluidised-bed (Pattiya, 2011) and free-fall (Pattiya et al., 2012) reactors, giving bio-oil yields of 63–65 wt.% (Pattiya, 2011) and 50 wt.% (Pattiya et al., 2012), respectively. This could indicate that the efficiency of the liquid bio-oil production, especially in heating rate or heat transfer terms, in the counter-rotating twin screw reactor was inferior to that in the fluidised bed reactor and was comparable to that in the free fall reactor.

3.2. Effect of biomass particle size on product distribution

To examine the effect of biomass particle size on product distribution, pyrolysis of cassava rhizome of particle size ranges of <0.250, 0.250–0.425 and 0.425–0.600 mm was performed at 650 °C using 7 L/min nitrogen flow at 1 bar. The result is depicted by Fig. 3. It can be seen that the maximum bio-oil yield of 43.05 wt.% could be obtained with the particle size of 0.250–0.425 mm. The size range gave minimum char yield. The biomass particle size of <0.250 mm gave very low bio-oil yield of 26.8 wt.%. This is expected to be related to the high ash content of the fine biomass (Pattiya and Suttibak, 2012) because the ash contained alkali and alkaline earth metals that could catalyse secondary cracking and repolymerisation of pyrolysis vapour, which would ultimately lead to higher gas and char yield on the expense of the bio-oil yield. The large particle size of 0.425–0.600 mm also gave lower bio-oil yield of 37.84 wt.% than that of the 0. 250–0.425 mm. This is possibly because the large particles possessed lower surface area for heat absorption and the inner part of the particles did not gain enough heat for pyrolysis reactor. Similar finding was previously reported by Pattiya and Suttibak (2012) when pyrolysis cassava stalk of <0.600 mm in a fluidised-bed reactor.

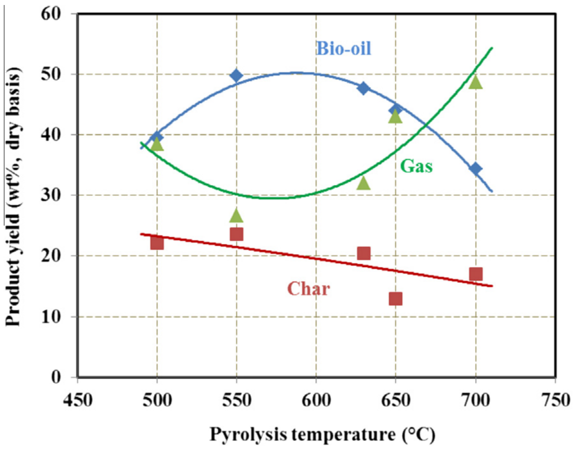

Fig. 2. Effect of pyrolysis temperature on product yields.

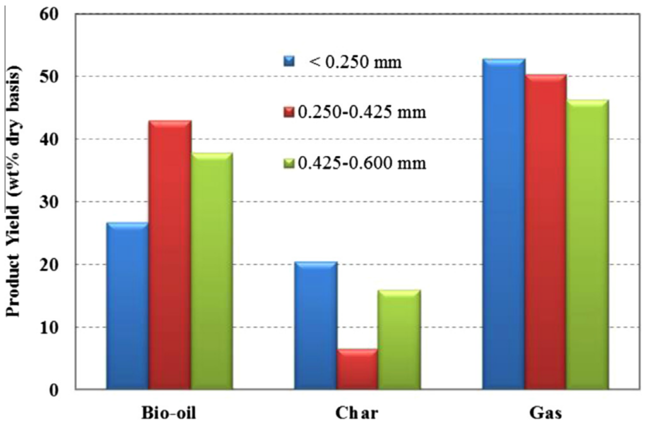

Fig. 3. Effect of biomass particle size on product yields.

3.3. Effect of heat transfer medium on product distribution

To observe the effect of heat transfer media, cassava rhizome of 0.250–0.425 mm was pyrolysed at 650 °C with a nitrogen flow rate of 7 L/min at 1 bar. The heat transfer medium was silica sand of 0.600–0.850 mm. It was found that a clear reduction of bio-oil yield took place with the experiment without the heat transfer media. This reduction occurred together with the increase in the char yield. The bio-oil yield decreased from 43.05 to 31.75 wt.% and the char yield increased from 6.56 to 17.05 wt.% when no silica sand was applied. It can also be noticed that the gas yields seem to be not affected by the presence (50.38 wt.%) or absence (51.2 wt.%) of the heat transfer medium. The reason why the heat transfer medium could increase the bio-oil yield and decrease the char yield in the twin screw auger reactor is due to the enhanced rate of heat transfer by the hot silica sand. Similar finding was reported by Brown and Brown (2012) that when increasing the mass flow rates of sand from 9 to 21 kg/h, the bio-oil yield was increased from 42.2% to 47.5%.

3.4. Effect of sweeping gas flow rate and pressure on product distribution

Nitrogen at 1 bar pre-heated to 400 °C was used as sweeping gas and entered the auger reactor at the top near the biomass inlet. The flow rate of nitrogen measured at room temperature varied from 4 to 7 L/min was supplied to the reactor which was controller at 650 °C. Fig. 4(a) shows the influence of the sweeping gas flow rate on pyrolysis product yield. The maximum bio-oil yield of 45.04 wt.% was obtained at the gas flow rate of 4 L/min. When increasing the flow rate from 4 to 10 L/min, the bio-oil yield steadily decreased. When the effect of nitrogen flow rate at 1.5–3.5 L/min was investigated by Brown and Brown (2012), it was found that the maximum bio-oil yield of 73 wt.% was obtained at the flow rate of 3.5 L/min.

Additional experiments were carried out at 2 and 3 bar to investigate the effect on sweeping gas pressure. Fig. 4(b) shows that the nitrogen pressure slightly affected the bio-oil yield and the maximum bio-oil yield of 43.2 wt.% occurred at 2 bar. Fig. 4(b) also shows that at 2 bar, the char yield was maximised, while the gas yield was minimised.

3.5. Characteritisation of bio-oil

The liquid bio-oil produced in this work was separated into organic and aqueous phases. Only the organic bio-oil from a selected run was characterised in terms of water content, solids content, density, heating value, pH and kinematic viscosity as summarised in Table 2 in comparison with literature data (Ingram et al., 2007; Pattiya, 2011; Şensöz et al., 2000).

The water content of the twin screw reactor bio-oil in this work was significantly lower than those from the free-fall (Pattiya et al., 2012) and fluidised bed (Pattiya and Suttibak, 2012) reactors when applying the same biomass type (cassava rhizome) and lower than those from the single screw reactor when applying pine wood and bark (Ingram et al., 2007) as feedstock. The low water content of bio-oil in this work could be explained by the fact that it was phase separated and the majority of water was not included in the organic phase. The phase separation can be regarded as an advantage since part of the unwanted water could be easily removed. Therefore, the bio-oil of low water content would be beneficial to several applications.

The solids content of bio-oil in this work was rather high (13.11%) compared to literature data (Ingram et al., 2007; Pattiya et al., 2012; Pattiya and Suttibak, 2012). This is expected to be due to the agglomeration of the highly viscous bio-oil to become solid gum. The gum may be destroyed by, for example, solvent dilution. This solids content reflects the high viscosity of the bio-oil (1618.9 cSt) compared to traditional bio-oil such as cassava rhizome bio-oil from a fluidised-bed reactor (51 cSt) or pine wood bio-oil from single screw reactor (60.9 cSt). It is therefore sug-

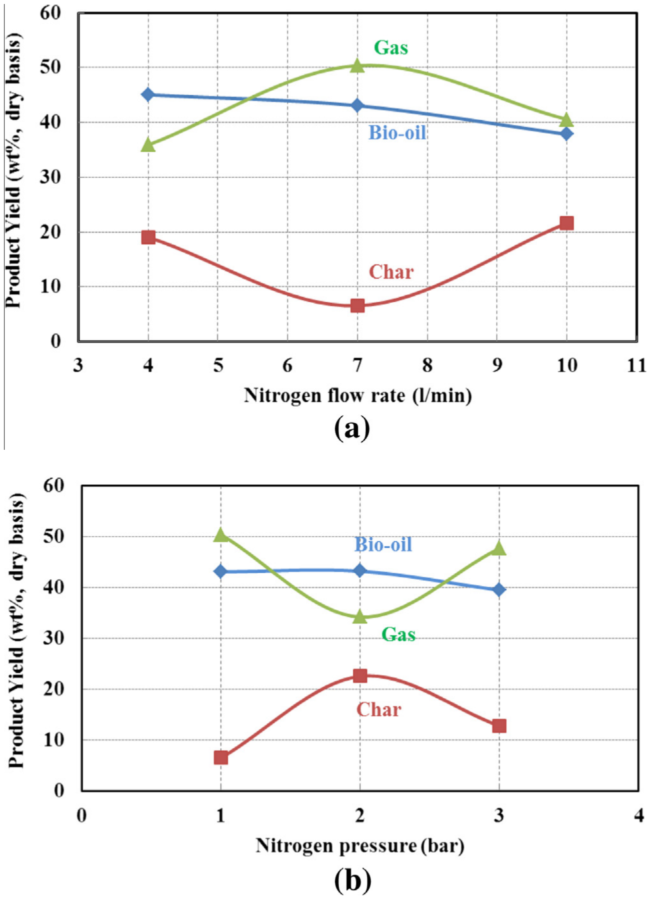

Fig. 4. Effect of sweeping gas flow rate (a) and pressure (b) on product yields.

Table 2
Characteristics of organic bio-oil.

	This work	Pattiya et al. (2012)	Pattiya and Suttibak (2012)	Ingram et al. (2007)	
Biomass	Cassava rhizome	Cassava rhizome	Cassava rhizome	Pine wood	Pine bark
Reactor	Twin screw	Free-fall	Fluidised-bed	Single screw	
Water content (wt.%)	10.3	24.1	18.0	16.0	19.8
Solids content (wt.%)	13.11	4.30	0.80	0.19	2.10
Density (kg/dm^3)	1.04	1.08	1.10	1.19	1.17
Heating value (HHV, MJ/kg)	28.45	26.70	26.90	21.9	18.3
pH value	2.90	3.50	3.10	3.10	3.20
Viscosity (cSt)	1618.93	–	51	60.9	–

gested that if the bio-oil from the present work is to be used as fuel, its viscosity needs to be decreased by some upgrading techniques such as solvent addition, emulsification or catalytic processing.

Regarding the density and pH value of the bio oil, Table 2 shows that these values are in the range of typical bio-oil. It can be noticed that the density of the bio-oil in this study (1.04 kg/dm^3) was slightly lower than those of the other studies (1.08–1.19 kg/dm^3), even though the solids content was a lot higher. This could be due to the high porosity of the char or solids in bio-oil, which reflected the lower density of the solids that could escape from the cyclone and hot filter. In addition, the pH value of the bio-oil produced in this work (2.90) was slightly lower than those produced from other studies. This implied that the bio-oil produced with the twin screw reactor contained a slightly larger proportion of acidic compounds such as acetic, formic or lactic acids.

The more interesting property of bio-oil is the heating value. It is apparent that the higher heat value (HHV) of bio-oil (28.45 MJ/kg) could be considered as high value. This was higher than the ones obtained from free-fall (Pattiya et al., 2012), fluidised-bed (Pattiya and Suttibak, 2012) and single screw reactors (Ingram et al., 2007).

4. Conclusions

Pyrolysis of cassava rhizome was performed in a counter-rotating twin screw reactor unit to investigate the effect of several parameters. The optimum pyrolysis temperature was 555 °C, which gave the maximum bio-oil yield of 50%. The biomass particle size of 0.250–0.425 mm was found to maximise the bio-oil yield. The use silica sand could increase the bio-oil yield. The appropriate nitrogen flow rate and pressure were 4 L/min and 2 bar, respectively. The bio-oil produced by the counter-rotating twin screw reactor had lower water content and higher solids content than those from other types of reactor such as free-fall and fluidised-bed.

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